

TOPICAL REPORT ACCO-58

FIELD WORK PERFORMED AT MONTICELLO, UTAH, INCLUDING
STOCKPILE AMENABILITY AND ION EXCHANGE

by

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July 20, 1954

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Classification changed to Unclass.
by authority of Trust letter 9/29/55
by N. Farrell date 10/4/55

Contract AT(49-1)-533

Atomic Energy Division
American Cyanamid Company
Raw Materials Development Laboratory
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Winchester, Massachusetts

72004

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A B S T R A C T

Bench scale leaching, pilot plant testing, and ion exchange investigations were made on samples of ore stockpiled at Monticello, Utah. Some testing of carbonate leach tailings was also completed. All ores were leached with varying amounts of sulfuric acid at various densities, temperatures, grinds, and times. Uranium extractions ranging from 92 to 98 % were obtained on all ores by acid leaching. Liquors from the ores were found amenable to ion exchange with some reservations. Stockpiles 6, 16, 19, 24, 29, and 28 were tested with the bulk of the work performed on various samples of stockpile 28. Stockpile 28 contains ore shipped by the Utex Mining and Development Company and their Lessees.

TABLE OF CONTENTS

	<u>Page</u>
I. INTRODUCTION	5
II. OBJECT OF INVESTIGATION	5
III. SUMMARY AND CONCLUSIONS	6
A. Carbonate Tailings	6
B. Stockpile 28 (Utex)	6
C. Stockpile 29 (Slick Rock)	8
D. Stockpile 19 (White Canyon)	9
E. Stockpile 24 (Temple Mountain)	9
F. Stockpile 6 (Miscellaneous High Lime)	10
G. Two-inch Column Exhaustion Studies	10
IV. TEST WORK PROCEDURE	13
A. Bench Testing	13
B. Pilot Plant Testing	13
C. Ion-Exchange Test Work	14
D. Operation of the Columns	15
V. TEST WORK	16
A. Carbonate Leach Tailings	16
B. Stockpile 28 (Utex Ores)	18
1. Bench Scale Leach Testing	18
2. Exhaustion Studies-Utex Liquors	27
3. Exhaustion Data	29
4. Elution Studies	33
C. Stockpiles 19, 24, 29, 6, 16	39
ACKNOWLEDGEMENT	42

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I. INTRODUCTION

The full work program as originally outlined for Monticello was designed to test carbonate leach tailings now available and those currently being produced. An expansion of the Monticello mill had been planned to utilize these tailings as part of the feed.

During the course of this program, it became evident that shipments by the Utex Mining and Development Company and their Lessees were increasing to a point where definite treatment methods would have to be worked out. At this time, the Winchester Laboratory assigned this work to the Monticello Field Group for immediate action. Work has progressed since then on various phases of the investigation.

Somewhat later, the expansion planned for the Monticello mill evolved into a complete new acid leach section, to be operated separately from the present carbonate circuit. Because of this decision, a series of amenability tests was started on all stockpiles that might eventually be treated in the acid plant. These tests were small scale for the most part and were designed to indicate whether the ores would respond to acid leaching and ion exchange. Since previous acid leach amenability tests had been made * on most of the stockpiles, testing was for the most part limited to determine the ion exchange characteristics of the liquors produced by acid leaching.

A few exploratory tests were made to determine settling and filtering rates and to check the possibility of recovery of vanadium from barren slimes and liquors.

II. OBJECT OF INVESTIGATION

The object of investigation was to determine optimum conditions for acid leaching of ores from various stockpiles at Monticello, and especially Stockpile 28, which was studied exhaustively both as to leaching and ion exchange characteristics. Results from this testing are being used, in conjunction with those obtained at the Grand Junction Pilot Plant, to develop a flow-sheet for the proposed Monticello acid leach plant.

* ACCO-30 "Preliminary Leaching Tests for the Extraction of Uranium from Various Monticello Stockpile Ores."

III. SUMMARY AND CONCLUSIONS

The results of the test work done on each ore and process studies during the course of this investigation may be summarized as follows:

A. Carbonate Tailings

Carbonate leach tailings from the Monticello mill tailings area were treated by the Galigher Company in a 5 ton acid pugging, curing, and percolation leach pilot plant. Liquors from this plant constituted the feed to the two-inch column plant.

Preliminary small column (25 ml) ion exchange test work on this liquor indicated very poor exchange characteristics. A series of tests were made to determine the best method of obtaining better resin loadings.

Two factors, a high sulfate ion concentration and low uranium content, appeared to cause the ion exchange difficulty. Several methods of lowering sulfate ion concentration were studied, and a method of lowering sulfate ion concentration while raising uranium content was devised. The best and cheapest method of lowering sulfate ion concentration involved the use of either limestone or hydrated lime. A flow sheet utilizing high lime ores from stockpiles was developed and this not only lowered sulfate concentration, but also increased uranium concentration in the liquor.

Two-inch column testing of liquors produced in the pilot plant and treated with limed showed uranium loadings of from 20 to 30 grams U_3O_8 per liter of wet settled resin. Average loading for the runs was 27.3 gms. U_3O_8 per liter. Recovery from solution was 96.73 % and final product grade, after precipitation of eluate with ammonia and calcining, was over 90 % U_3O_8 .

Small column testing of liquors produced by pugging and curing of tailings and contacted with fresh high lime ore, showed saturation loadings of 79.7 grams U_3O_8 per liter of wet settled resin.

Three to five minute retention times were optimum for most of the liquors produced from tailings. Further work on tailings was suspended when the program on Utex ore was started. The work done by the Monticello Field Group indicated that acid leaching and ion exchange will at best be marginal when treating tailings.

B. Stockpile 28

Several different samples of ore were taken from Stockpile 28 and used in the test work program. These samples assayed from 0.38 to 0.57 % U_3O_8 , 0.75 to 1.25 % V_2O_5 , and from 1.20 to 17.0 % $CaCO_3$. Main uranium minerals are uraninite and carnotite.

Bench scale leaching tests, using sulfuric acid, showed that uranium recoveries of over 95 % were possible at proper acid concentrations using either an aqueous type leach or pugging and curing techniques. The vanadium extractions, in the aqueous leach, ranged from 50 to 75 % depending upon the amount of acid used.

Vanadium extractions of 90 % or over were obtained when the ore was pugged with 700 pounds of H_2SO_4 per ton of ore, or more, and cured for four days at ambient temperatures or for twenty hours at $100^\circ C$. Similar vanadium extractions were possible using the same amount, or slightly less acid in combination with hot ($85^\circ C$) leaching at 50 % solids with the addition of 10 to 20 pounds of sodium silico-fluoride per ton of ore.

Test work indicated that grinding of the ore beyond 30 mesh was unnecessary and that good extractions were possible with minus 16 mesh ore.

Optimum conditions for acid leaching Stockpile 28 were selected from a series of tests conducted jointly with the Galigher Company. The following are the conditions:

1. Acid - 700 pounds per ton of ore
2. Agitation Time - 12 hours
3. Grind - Minus 16 Mesh
4. Density - 50 % Solids
5. Temperature - $85^\circ C$.

The above conditions were selected on the basis of maximum uranium and vanadium recovery. Further test work disclosed that using less acid (down to 450 pounds per ton) would still give high uranium extractions, but that recovery of vanadium dropped to less than 50 %.

Preliminary settling tests were made on leach pulp produced under the optimum conditions. Very poor settling of the minus 16 mesh ore was observed without settling aid addition. Leach pulp was diluted to 3.0 to 1 before thickening. Final density was 45 % solids when 0.5 pound of Guartec per ton of ore was added and satisfactory settling was obtained in cold solution. Area requirements when using Guartex were about 2.0 square feet per ton per 24 hours. The pulp entered compression at relatively low density and the zone of free settling was rather narrow. However, test work indicated satisfactory rates were obtainable by the use of gum additives.

Ion-exchange of Utex acid leach liquors was tested in a standard four column two-inch column set-up with two columns on exhaustion and one on elution. The freshly eluted column was used as a scavenger unit.

Feed liquors to the columns averaged from 1.0 to 1.4 gms. U_3O_8 per liter and were treated with hydrated lime to raise the pH to 1.4 to 1.8. Redox potential of the liquors was generally below - 400 m.v. (oxidized) and no reduction was required.

Through June 7, 1954, 69 1/2 complete resin cycles (278 loading cycles) had been run in the columns using Utex liquors produced in the Galigher Company Pilot Plant.

Average retention time was 30 minutes and resin loadings were over 60 grams U₃O₈ per liter of wet settled resin. Recovery from solution averaged 99.8 % or better, for all cycles completed.

Elution of the columns was investigated with a variety of eluting solutions. The bulk of the work was done with a standard 0.9 M NH₄Cl - 0.1 M HCl eluant, recycled with chloride make-up, and acidified to pH 1.0 with sulfuric acid. Average elution retention time was 12.4 mins. Average eluate grade was 7.33 grams U₃O₈ per liter, and final eluates were about 0.15 grams U₃O₈ per liter.

Anhydrous ammonia was used to precipitate eluates, and about 0.7 pound per pound of U₃O₈ was required. Eluate make-up required about 2.0 pounds of 36 % HCl and 3.0 pounds of 98 % H₂SO₄ per pound of uranium eluted.

Utex ore currently stockpiled at Monticello was amenable to acid leaching and ion exchange. The question of column operation vs. resin-in-pulp was not studied here. Data for such a study is available, however. The nature of the ore suggested that a solid-liquid separation would be difficult to achieve. If clear liquor was used for ion exchange, a C.C.D. system would be the easiest way to make the separation. Filtering characteristics of the ore in acid leach pulps were poor in all instances.

Vanadium extractions of over 90 % were obtained from this ore by hot, aqueous leaching without pugging or curing. The cost of using additional acid on Utex ore for the extraction of vanadium was too great to be economical. Previous experience at Monticello with pugged and cured ore indicated that the techniques employed there were not adaptable to large scale work.

The buildup of molybdenum on the resin required a periodic cleanup. Removal of this molybdenum contamination would be necessary about twice a year under normal plant conditions.

The grade of the final product depended on the type of elution used. Ammonium chloride eluate produced a calcined product which ran 94 % U₃O₈, small amounts of molybdenum, and about 1 - 1.5 % P₂O₅. Elution with sodium chloride produced a product which contained about 74 % U₃O₈ with sodium being the chief impurity.

C. Stockpile 29 (Slick Rock)

Leaching was carried out at 85°C. on 400 pound batches of 10 mesh ore at 50 % solids using 300 pounds sulfuric acid per ton. The leach liquors were diluted to give a solution to solids ratio of 3 to 1 and a head assay of 1 - 1.5 grams U₃O₈ per liter. The pH was adjusted to 1.5 with lime and filtered prior to ion exchange.

Variations in dilution were used to obtain differences in uranium content and to determine loading characteristics. Head variation, between 0.96 and 1.53 gms. U_3O_8 /liter, did not affect loadings.

Exhaustion-retention times were varied from 2.0 to 5.6 minutes with little or no change in loadings. Optimum retention time was 3.5 minutes.

Elution of loaded columns was made with fresh 0.9M NH_4Cl - 0.1M HCl for each column. An average of 18.7 column volumes of eluate was required for complete elution. Eluates averaged 3.53 gms. U_3O_8 /liter. Final product grade, after ammonia precipitation and calcining ran from 91.4 to 93.4 % U_3O_8 .

Average resin loading for 10 complete cycles was 65.5 gms. U_3O_8 /liter w.s.r. No drop off in loadings was noticed during the 10 cycles. Recovery from solution was 99.64 %.

Slick Rock ores make up a large part of the material stockpiled at Monticello. The ores are generally carnotites and have previously been treated by salt roasting and carbonate leaching. Testwork here has shown them to be amenable to acid leaching and ion-exchange. The testing on this ore was mainly to determine ion-exchange amenability. Further testing of the ore should be considered, especially as to optimum leach conditions and settling and filtering characteristics.

D. Stockpile 19 (White Canyon)

Stockpile 19 was leached in a manner similar to that used for Stockpile 29. Used in the leach were 300 pounds of sulfuric acid per ton. Head solution for ion-exchange was prepared in accordance with standard practice except that calcium carbonate was used for neutralization. Head analysis was 1.0 gms. U_3O_8 /liter, 0.36 gm. V_2O_5 /liter and 1.15 gms. Cu /liter at a pH of 1.5 and emf of -380 mv.

At exhaustion-retention times of 3.0 to 6.0 minutes, resin loadings were about 60 grams U_3O_8 /liter w.s.r. and recovery from solution was about 99.5%. Elution with 1.0 molar NH_4Cl acidified to pH 1.0 with H_2SO_4 required an average of 18 column volumes. Elution of the columns was slow and incomplete, and split elution would probably help with this liquor.

Resin loadings with this liquor dropped off to 50 grams per liter after three cycles and remained at that figure for the balance of the ten cycles.

A run in the two-inch columns using stockpile 19 liquors was planned for further study.

E. Stockpile 24 (Temple Mountain)

Calcined Temple Mountain ore was obtained from the Vitro Chemical Company through the U. S. Bureau of Mines. This calcined product, minus 10 mesh, was leached batchwise as were those previous. Leaching was accomplished

using 300 pounds of sulfuric acid per ton of ore. Leach liquor was diluted and neutralized in the standard manner. Head analysis of the feed liquor to the 1/2-inch unit was 0.90 gms. U_3O_8 /liter, 2.84 gms. V_2O_5 /liter, at pH 1.65 and -410 m.v.

Exhaustion retention times of from 2.6 to 5.2 minutes gave resin loadings averaging 64.2 gms U_3O_8 /liter w.s.r. Elution of the columns with standard 0.9M NH_4Cl - 0.1M HCl was complete, but rather slow. An average of 20.5 column volumes was required to obtain complete elution. Ten cycles were run on this liquor and no appreciable drops in loading were noticed. The testwork done in Monticello indicated that this liquor appears to be amenable to ion-exchange.

F. Stockpile 6 (Miscellaneous High Lime)

The half-inch column work on this liquor is now in progress but no definite results have as yet been obtained.

G. Two-inch Column Exhaustion Studies

Exhaustion data for the three types of solutions showed from 5 to 10 grams U_3O_8 per liter difference in the loading characteristics of the liquors. Apparently, cold, aqueous leaching with only enough acid present to extract the uranium values gave the best liquor for ion-exchange. Cold leach liquors are now being run in the columns and loadings appear to be holding near 70 grams U_3O_8 per liter of wet settled resin. Loadings for both pugged and hot leach liquor averaged from 60 to 65 grams U_3O_8 per liter of wet settled resin.

Loading characteristics of pug liquor at a final dilution of 2.5 to 1 and a 5 minute retention time, and of hot leach liquor at a final dilution of 3.0 to 1 and a 3 minute retention time are shown in Figures 1 and 2, respectively. In both cases the final column is saturated when the second column reaches cutoff.

All types of liquors treated exhibited good ion-exchange characteristics and high loadings. The pH of the IX feed must be held within the limits of from 1.4 to 1.8 and redox potential of solutions should be below minus 400 millivolts. The best results were obtained when the uranium content of the feed liquor was held at from 1.0 to 1.5 grams U_3O_8 per liter. Most leaching and solid-liquid separation methods now under consideration will deliver feed liquor in this range from current Utex ores.

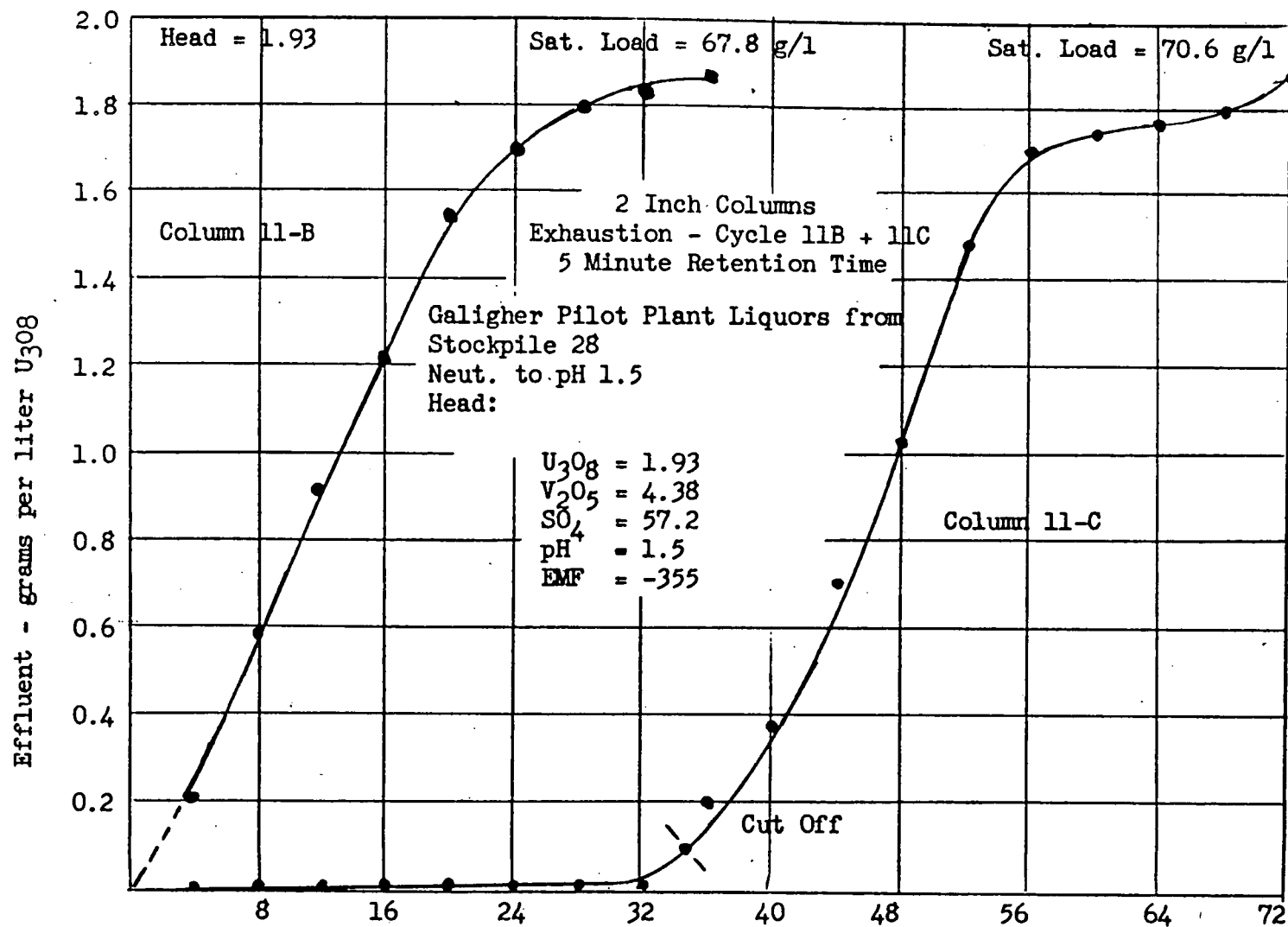


Figure 1 - Effluent - Column Volumes

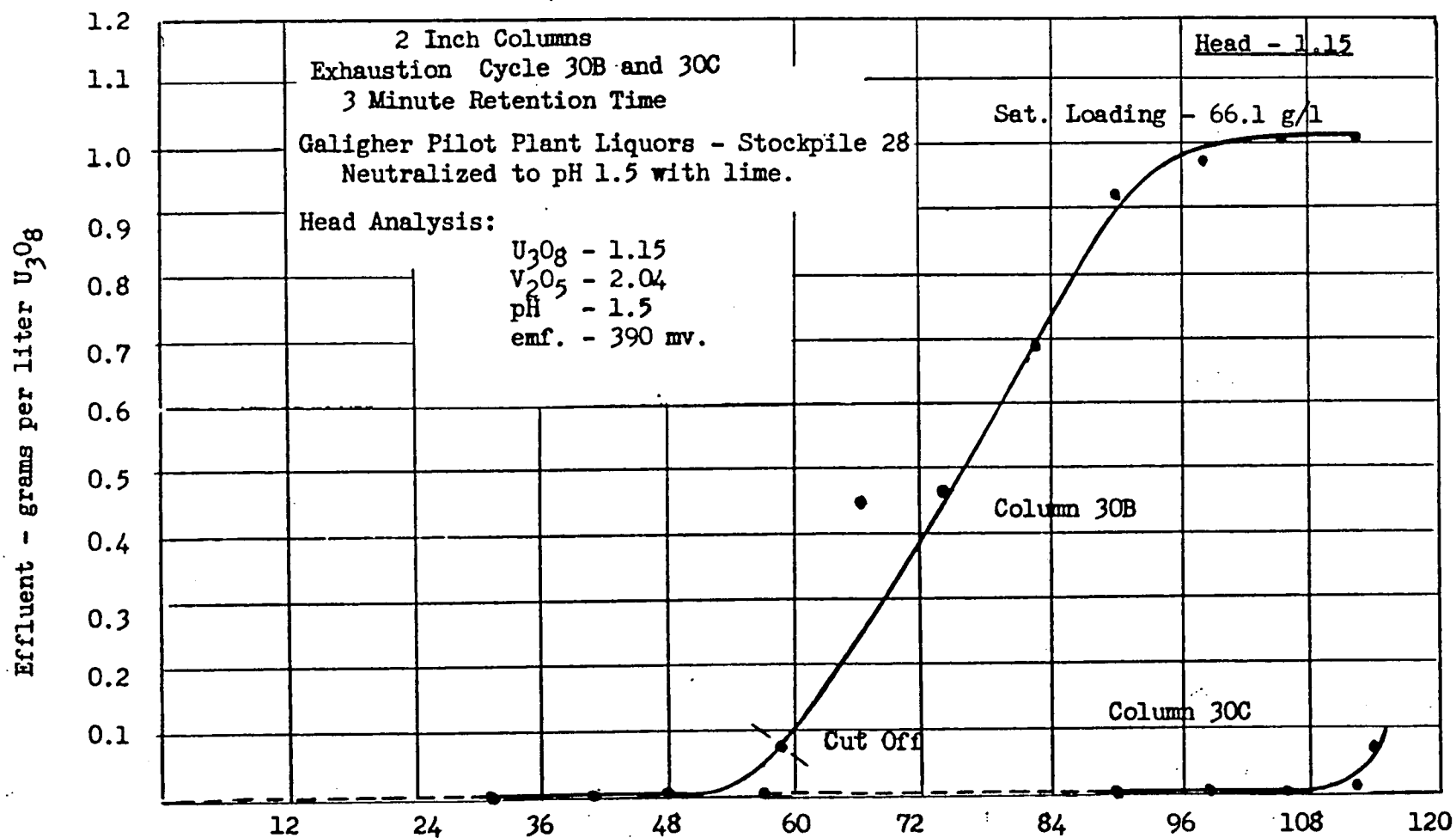


Figure 2 - Effluent - Column Volumes

IV. TEST WORK PROCEDURE

A. Bench Testing

Aqueous leach tests were conducted with 500 gram ore charges. Sufficient water was added to this charge to achieve the desired density and the specified amount of acid and other reagents were added. The leach pulp was agitated by means of motor driven stirrers and heat, if used, was supplied by electric hot plate.

Pulps were filtered on Buchner funnels and filter cakes were washed with 1-150 ml 5% acid wash and 2-150 ml water washes. In most cases, solutions were not saved, and extraction figures based on head and residue assays were used.

Pugging of the ore was done in enamel sample pans. One thousand grams of ore (usually minus 10 mesh) was thoroughly mixed with 200 ml of water. The acid was then added and the ore again thoroughly mixed. During the cure period, the ore was covered and periodically sprayed with water to keep it moist. When the ore was allowed to dry completely, it set up and was difficult to remove from the pan. Agitation of the cured material was at 50 % solids, room temperature, for the time specified. To the leach was added 0.5 pound Quartec per ton of pulp prior to filtration and the cake was washed with three water washes and acidified to pH 1.5 with sulfuric acid. Final dilution, including washes, was 2.5 to 1.

Settling tests were conducted according to a modified Coe and Clevenger method as published in the "Denver Equipment Index".

Filtering tests were made using a standard 0.1 sq. ft. Oliver test leaf, fitted with an orlon cloth.

B. Pilot Plant Testing

The pilot plant used for leach testing of Utex ores encountered in this study was adapted from the acid pugging and curing plant used to develop material from the Galigher Company tailings. Originally, the plant was designed and operated as a pugging and curing unit, with percolation leaching of the ore following the cure period with agitation leaching and filtration.

Pilot plant flow started with a small storage bin to which minus 10 mesh ore from the main crushing plant was delivered by truck and hand labor. A belt feeder under the storage bin fed ore to a 2 1/2 x 4 foot horizontal cylindrical mixer equipped with spiral mixing blades and mechanical knockers to prevent ore buildup. Ore from the pugger was dropped into a 5 ton bin where it was allowed to cure. After curing, the ore was drawn off from the bin by belt conveyor and delivered to the agitator, a 30 inch by 4 foot rubber covered tank with a 10 inch rubber covered mild steel impeller. Pulp from the agitator was filtered directly on two 4 foot diameter Bee Tee tilting filters. Solution from the filters was pumped to storage for use in ion-exchange test work.

On tests where pugging and curing was used, agitation was done batchwise. This occurred because the material in the cure bin set up so completely that bottom discharge was impossible. In several instances, the material had to be broken up by bars and then shoveled into the agitator tank by hand.

The pilot plant was modified further to allow aqueous leaching, both continuous and batchwise. Heat could be added to the agitator tanks by means of steam to achieve temperatures required in hot acid leaching. After the campaign on pugging and curing was completed, hot acid leach testing was started varying the amounts of acid, contact time, and temperatures. In general, hot acid leaching practice in the pilot plant followed the optimum conditions that were found in the laboratory bench work.

Two continuous leach tests were performed in the pilot plant feeding 6 tons of ore every 24 hours. These tests were inconclusive however, as mechanical failure of agitator blades forced a shutdown.

The above discussion deals with pilot plant practice in a general way only. Specific mention of test results would be cumbersome and since ion-exchange test work was not tied into pilot plant recovery, any results presented would have little meaning. This brief outline has been included to indicate the source of liquors used for ion-exchange testing in the two-inch columns. The Galigher Company was for a time performing pilot plant tests to secure data for the proposed operating plant. When this work was completed to their satisfaction, the pilot plant was placed on intermittent operation and used merely to supply liquor for ion-exchange testing when the need arose.

It may be stated that the Galigher Company found that all ores responded in the pilot plant as bench work indicated it would. Aside from mechanical difficulties encountered in pugging and curing large amounts of ore (2 to 5 tons) and the failure of rubber covered agitator blades due to cuts caused by tramp materials in the pulp, pilot plant operation and recovery was satisfactory.

C. Ion-Exchange Test Work

A standard four column, two inch diameter ion-exchange unit was installed to treat the acid leach liquors produced in the Galigher Company pilot plant.

Each column contained 1.5 liters of Amberlite IRA-400 resin. The columns could be operated with either two or three columns in series on the loading cycle and the remaining one or two columns on the elution cycle.

A 3/8-inch Ecco pump was used to transfer liquor from storage through the columns. Recirculation to the storage tank allowed pressure control to be maintained so that no damage was done to the valves or columns. A continuous drip sampler was placed in the barren line from the columns in order to obtain a barren composite sample.

The eluting solution flow was by gravity feed. Makeup eluant was pumped up to elution storage by evacuation of the storage bottle. Water was supplied to the columns through a pressure regulator valve set at 5 p.s.i.g.

Leach liquor from the pilot plant was transferred to a 4 foot rubber lined tank with an attached agitator for pH adjustment. After the precipitated gypsum settled out the liquor was transferred to another 4 foot tank for storage. A sand filter was used to clarify the liquor for part of the test work program but if care was exercised when drawing out the liquor, no filter was needed.

Precipitation of the eluate was carried out in an earthenware crock using anhydrous ammonia. The yellow cake product was filtered on Buchner funnels after first settling and drawing off clear barren eluate. Calcination of the product was done in an oil fired muffle furnace. The final product was weighed, assayed and reserved for accountability.

D. Operation of the Columns

Two columns were operated in series to the cutoff point on the exhaustion cycle. This cutoff point was arbitrarily designated as the point at which the effluent from the second column contained from 2 to 3% of the uranium in the feed liquor. As a safety factor a third column was put in series on exhaustion before cutoff was reached. In normal operation a retention time is selected such that the first column reaches saturation when the second column reaches the cutoff point.

During the exhaustion cycle, the specified flow rate was maintained by means of a flowmeter. Most of the work was done at a 3.0 minute retention time, which, with 1500 ml of resin per column, corresponded to a flow of 200 ml per minute. With liquors containing from 1.0 to 1.4 g U_3O_8 /L cycle times for exhaustion were of the order of 7 to 8 hours.

As a general rule, exhaustion retention time was dictated by three factors. These were: (1) The head analysis of the feed liquor; (2) Elution rate and total elution time, including column preparation; and (3) Resin loadings. Most of the liquor was passed through the columns at 3.0 minute retention time. This resulted in cycle times of about 7 to 8 hours with liquors diluted 3.0 to 1, with a U_3O_8 content of from 1.0 to 1.4 grams per liter.

In actual plant practice, the exhaustion cycle would be run on a time basis. The length of time selected for loading would be something slightly more than that required to reach cut-off and on the average, loading would go to saturation in the first column. The third or scavenger column would take care of any variations in loading and would maintain recovery.

V. TEST WORKA. Carbonate Leach Tailings

The two-inch unit was operated on acid leach liquors produced from pugging and curing carbonate leach tailings from July 9 to August 28, 1953.

Five different batches of solution were drawn off for column testing. Each batch was a mixture of pilot plant liquors from any one testing program. Liquors from the pilot plant were neutralized with lime to pH 1.5. Original pH varied from 0.4 to 1.0. After neutralization, precipitated gypsum was allowed to settle and solution was decanted through a sand filter to IX storage. Solutions used and assays are presented in Table I. Designator "A" or "B" indicates treated solution, either by neutralization or reduction.

TABLE I

Tailings Leach Liquors used in IX Plant

Head	IX	Assay g/l				
No.	Cycle Nos.	U ₃ O ₈	V ₂ O ₅	SO ₄	pH	emf
4	1A- 2A	.711	4.30	150.3	.85	--
5	----	.64	3.81	140.9	1.0	--
5A	2B- 4B	.65	3.81	125.3	1.5	--
6	----	.75	4.22	93.4	.45	-680
6A	4C- 5B	.58	4.02	71.6	1.5	-640
6B	5C- 6A	.50	4.02	71.6	1.5	-450
7	----	.53	3.91	83.5	1.0	-425
7A	6B- 7C	.51	3.91	76.4	1.5	-425
8	----	.53	3.91	83.5	1.0	-425
8A	7D- 10A	.52	4.03	75.9	1.5	-425

Heads 4 and 5 were produced from high-acid runs in the pilot plant. Head 6 was produced with less acid and sodium chlorate. Heads 7 and 8 were produced from fluosilicate pugs with lower amounts of acid.

All solutions treated were loaded to 25 gms. per liter of resin or better. Elution of the loaded columns was satisfactory and complete within 8 to 10 column volumes. Various eluate make up schemes were tried, including bleed. The best was a 10 to 20 percent bleed, followed by makeup with NH₄Cl and HCl to pH 1.0.

Final product analysis for a composite sample from the runs on tailings are presented in Table II.

TABLE II

Final Product from Tailings Liquor

Assay, percent

<u>U₃O₈</u>	<u>V₂O₅</u>	<u>Cl⁻</u>	<u>S⁻</u>	<u>Cu</u>	<u>P₂O₅</u>	<u>Fe</u>
90.71	.40	tr	<.05	<.05	1.65	2.70

Uranium balance for the cycles completed on Tailings liquor is shown in Table III. No attempt was made to balance liquors as received from the Galigher Company, as excess liquors, along with the settled gypsum were sent to tails.

TABLE III

IX Plant Balance - Tailings Liquor

U ₃ O ₈	Feed to IX Columns	1063.0 grams
U ₃ O ₈	Contained in Backwash	1.0
U ₃ O ₈	Contained in Column Effluent	34.8
U ₃ O ₈	Absorbed by Columns	1027.2 grams
U ₃ O ₈	Contained in Eluates	1006.2
U ₃ O ₈	Unaccountable - (Recycle, etc.)	21.0 grams

Tailings liquor as produced in the pilot plant was neutralized with fresh, high lime ore from stockpile 16, and the resulting liquor was tested in small columns. Table IV shows the assay values of a liquor treated this way. The pug liquor was adjusted to pH 2.2 using stockpile 16 ore, reduced to -390 mv with iron, and diluted 50 percent.

TABLE IV

Liquor Neutralized with High Lime Ore

Assay, g/l

	<u>U₃O₈</u>	<u>V₂O₅</u>	<u>Fe</u>	<u>SO₄</u>	<u>Cl</u>	<u>pH</u>
Original Pug Liquor	0.29	3.04	11.1	152.0	1.97	0.4
*Neutralized Liquor	1.14	2.85	10.0	93.1	----	2.2

*Extraction from stockpile 16 was 85 percent.

Ion-exchange of this liquor, at 3 minutes retention time, resulted in a breakthrough loading of 27.4 mg./ml. and saturation loading of 57.7 mg./ml.

When retention time was increased to 6 minutes, breakthrough loading increased to 41.0 mg./ml. and saturation loading to 70 mg./ml.

As testwork progressed on tailings liquor, a definite trend showed that loadings were dependent on the sulfate to uranium ion ratio. This is illustrated in Table V.

TABLE V

Resin Loadings vs. Sulfate: Uranium Ratio

IRA 400 Resin

Test No.	SO ₄ to U ₃ O ₈ Ratio, g./L.	pH	Loadings		Remarks
			Breakthru	mg. U ₃ O ₈ /ml. w.s.r. Saturation	
2 -A	146	0.7	5.0	----	
1 -A	146	1.5	11.6	21.4	emf= -350 mv.
1 -B	146	1.0	3.0	----	emf= -350 mv.
2 -B	610	1.6	5.0	27.0	not reduced
2 -A	88	1.7	50.0	79.7	not reduced
3 -B	88	1.8	59.0	80.7	emf= -420 mv.
3 -A	82	2.2	27.4	57.7	emf= -390 mv.
4 -A	82	2.2	41.0	70.0	Same as 3-A except double retention time
4 -B	97	1.0	15.7	31.5	emf= -420 mv.
5 -A	162	1.4	15.6	27.1	not reduced
5 -B	286	1.1	4.3	13.8	not reduced
6 -A	141	2.3	15.0	----	not reduced
7 -A	268	1.0	4.8	----	not reduced

The tests shown in Table V were made on a variety of liquors. It is interesting to note that the highest loadings were obtained on liquors where the sulfate to uranium ratio was below 100. Acceptable loadings were obtained when the ratio went to 150 if the pH were also raised above 1.5.

B. Stockpile 28 - Utex Ores

1. Bench scale leach testing

Preliminary testing of the ore was confined to aqueous leaching and pugging of the ore, with varying amounts of sulfuric acid, oxidants, and other reagents. Since the ore contained a high percentage of calcium carbonate, testing at Monticello was confined to acid leaching.

Utex ore from the Mi Vida claim is a fine-grained uranium bearing sandstone. Various samples were taken from the stockpile at Monticello and from sample rejects from the American Smelting and Refining Company sampling plant at Monticello.

In general, samples of the ore analyzed as follows:

<u>%U₃O₈</u>	<u>%V₂O₅</u>	<u>%CaCO₃</u>
.38 - .57	.75 - 1.25	12.0 - 17.0

According to the U. S. Bureau of Mines*, microscopic examination of the ore shows the main constituents to be quartz, clays, and calcite. Small amounts of pyrite, galena, and asphaltic material are present. The uranium is present as carnotite and uraninite, almost evenly divided. Evans states, "Liberation of a large proportion of the carnotite will require grinding the ore to pass a 150 mesh screen, but considerably finer grinding to slime sizes will be required to free the uraninite from the clay."

Analyses of all head samples encountered and used in the test work program at Monticello are presented in Table VI, shown below. Preliminary testing was done with Head number VI, which consisted of a sample taken from moisture rejects from the sample plant.

TABLE VI

Head Analyses - Utex Ore Samples

Head No.	Use	Assay, %					
		<u>U₃O₈</u>	<u>V₂O₅</u>	<u>CaCO₃</u>	<u>MgO</u>	<u>SiO₂</u>	<u>Cu</u>
1	Bench Testing	.57	1.25	14.46	1.65	68.47	.016
3	Pilot Plant	.37	.93	15.83	MgCO ₃	--	--
*4	Bench Testing	.390	.75	17.67	3.22	66.80	--
6	Pilot Plant	.427	1.11	--	--	--	--

*This sample was split three ways and duplicates were sent to USBM, Salt Lake and to Winchester.

Results of preliminary aqueous leach tests are presented in Table VII.

*T.M.E. 339-1, A.E.C. 211, Report of Microscopic Examination of a Uranium Ore Shipped to Monticello, Utah, by the Utex Mining and Development Company. By LeMar G. Evans, 8 July, 1953.

TABLE VII

Aqueous Leaching of Stockpile 28 Ore - Head No. 1

Conditions: minus 10 mesh ore 500 g charge
 50% solids
 Room temperature
 Leach time - 4 hours
 3-150 ml water washes

Test No.	Acid* Lb/T	Preg Liq g/L		Wash Liq g/L		Residue %		Recovery %	
		U ₃ O ₈	V ₂ O ₅	U ₃ O ₈	V ₂ O ₅	U ₃ O ₈	V ₂ O ₅	U ₃ O ₈	V ₂ O ₅
1	300	4.90	3.85	1.16	1.03	.125	.90	80.3	31.3
2	350	4.64	3.89	1.16	1.06	.108	.90	81.6	31.6
3	400	4.90	4.28	1.12	.94	.079	.86	86.7	34.2
4	500	4.87	4.49	1.34	.95	.090	.84	85.1	34.8

* Pounds 100% acid.

As indicated from these tests, a maximum recovery of uranium on minus 10 mesh ore was obtained at 400 pounds of acid per ton. Vanadium recovery was low on all tests. To improve vanadium recovery, a series of tests using the so-called "pugging" technique was made. Results of these tests are presented in Table VIII.

TABLE VIII

Pugging, Curing and Agitation of Stockpile 28 Ore - Head No. 1

Test No.	Reagents Lb/T		Cure Time, Hours	Agitation Time	Residue, %		Extraction, %	
	H ₂ SO ₄	NaClO ₃			U ₃ O ₈	V ₂ O ₅	U ₃ O ₈	V ₂ O ₅
5-L	350	--	16	40 min	.087	.76	88.4	39.2
6-L	350	--	40	Percolation**	.092	.89	85.5	28.8
7-L	400	10	23	40 min	.047	.62	90.1	52.2
8-L	400	20	23	3 hr	.073	.56	86.8	56.2
9-L	400	--	18*	4 hr	.082	.44	87.1	68.8
10-L	500	--	18	4 hr	.045	.78	92.1	43.9
11-L	500	10	18	4 hr	.085	.66	85.1	48.8
13-L	500	--	4 days	4 hr	.023	.54	95.9	58.9

* Cured at 115°C until dry.

** 1000 ml water passed through ore bed twice, then cake washed.

Pugging of the ore was done in enamel sample pans. A sample of 1000 grams of the ore was thoroughly mixed with 200 ml of water. Acid was added and the ore was again thoroughly mixed. During the cure period, the ore was covered and periodically sprayed with water to keep it moist.

When the ore was allowed to dry completely, it set up and was difficult to remove from the pan. Agitation of the cured material was at 50% solids and room temperature for the time specified. To the leach pulp, 0.5 pounds of quartec per ton was added prior to filtration and the cake was washed with 3 water washes and acidified to pH 1.5 with sulfuric acid. Final dilution, including washes, was 2.5 to 1.

Results of pugging and curing suggest that an oxidant improves extraction to some extent. It is evident, also, that either a long cure time is necessary, or a short cure at an elevated temperature is required. Percolation of the minus 10 mesh ore might be possible from observation of tests. The ore filters very poorly even with the addition of glue and quartec, but settles quite well with the addition of quartec.

Hot acid leaching of the ore was initiated in August, 1953. Preliminary test results are presented in Table IX.

TABLE IX

Aqueous Leaching - Stockpile 28 Ore - Head No. 1

4 Hour Leach - 50% Solids

Test No.	Acid Lb/T	NaClO ₃ Lb/T	Leach Temp °C	Residue, %		Extraction, %	
				U ₃ O ₈	V ₂ O ₅	U ₃ O ₈	V ₂ O ₅
14-L	500	--	70	.031	.60	93.9	53.2
15-L	500	10	70	.025	.45	96.3	65.2
16-L	500	--	90	.027	.48	95.6	66.4
17-L	500	10	90	.022	.40	96.4	69.6
19-L*	500	--	90	.027	.55	95.5	59.5
20-L	600	--	90	.021	.43	96.5	68.5
21-L**	500	--	90	.015	.48	97.7	69.2
22-L	400	--	90	.031	.64	94.6	50.0
24-L	800	--	90	.012	.30	97.6	76.0***

*Leach diluted to 2.5 to 1 prior to filtering

**Test 21-L leached 8 hours

***Recovery based on head and residue assay

It might be noted here that all tests up through 22-L were complete tests, including assay of leach and wash liquors. "Extraction" figures given for these tests are actually "recovery" figures, based on a calculated head assay. Tests subsequent to 22-L were calculated on an "extraction" basis, using head and residue assays.

Results of these preliminary tests plus some preliminary testing by the Galigher Company, led to a joint investigation into some of the variables encountered. These tests were made using Head Sample number 4, which was also the sample sent to other laboratories. Results of

these tests were presented in letters from H. A. Johnson to John L. Chapman dated October 30 and December 11, 1953. This test work covered hot acid and aqueous leaching. The pugging and curing work was being handled by the Bureau of Mines at that time.

Test results are presented in Tables X through XVI. Acid amounts given are for pounds of 96% acid. *Denotes Galigher Company tests.

TABLE X

Variable - Acid Requirements

Constants: Grind - minus 16 mesh
Density - 50% solids
Temperature - 85°C
Agitation Time - 6 hr

Test No.	H ₂ SO ₄ lb/T	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
25-L	400	82.8	42.6
26-L	500	90.5	56.0
*574	600	94.4	64.0
*573	700	98.2	69.3 (Optimum U ₃ O ₈)
*572	800	97.9	78.7

TABLE XI

Variable - Agitation Time

Constants: Acid - 700 lb/T
Grind - minus 16 mesh
Density - 50% solids
Temperature - 85°C

Test No.	Agitation Time, Hr	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
*615	1.5	85.6	49.3
*575	3.0	93.1	66.7
*576	6.0	97.7	72.0 (Optimum U ₃ O ₈)
*590-A	9.0	96.4	77.3
*578	12.0	96.7	82.7
*616	20.0	93.6	82.7

TABLE XII

Variable - Grinding

Constants: Acid - 700 lb/T
 Temperature - 85°C
 Density - 50% solids
 Agitation time - 6 hr

Test No.	Grinding U.S. Mesh	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
*605	10	94.1	73.3
*573	16	98.2	69.3 (Optimum U ₃ O ₈)
*606	20	96.7	70.7
*609	30	97.9	76.0
*607	40	95.9	77.3
*608	50	97.7	74.7

TABLE XIII

Variable - Density

Constants: Acid - 700 pounds per ton
 Grind - Minus 16 mesh
 Temperature - 85°C
 Agitation Time - 6 hr

Test No.	Density % Solids	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
27-L	30	96.2	60.0
*593A	35	96.0	65.3
*594A	40	96.7	66.7
*595A	45	93.8	72.0
*573	50	98.2	69.3 (Optimum U ₃ O ₈)
*618	55	96.9	80.0
*619	60	93.9	69.3
*617	65	84.9	82.7

TABLE XIV

Variable - Agitation Temperature

Constants: Acid - 700 pounds per ton
 Grind - minus 16 mesh
 Density - 50% solids
 Agitation time - 6 hr

Test No.	Agitation Temp, °C	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
30-L	30	84.1	29.3
31-L	60	95.1	49.5
32-L	70	97.2	57.4
*573	85	98.2	69.3 (Optimum U ₃ O ₈)
*629	90	96.9	82.7

TABLE XV

Variable - Effect of Oxidant

Constants: Acid - 700 pounds per ton
 Grind - minus 20 mesh
 Density - 50% solids
 Agitation Temperature - 85°C
 Agitation Time - 6 hr

Test No.	NaClO ₃ lb/T	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
*573	none	98.2	69.3 (Optimum U ₃ O ₈)
*610	5	97.2	77.4
*611	10	97.4	85.3
*612	15	97.4	82.7
*613	20	96.4	77.4

TABLE XVI

Variable - Effect of Roasting

Constants: Acid - 700 pounds per ton
 Grind - minus 16 mesh
 Density - 50% solids
 Agitation Temperature - 85°C
 Agitation Time - 6 hr
 Roasting Time - 2 hr

Test No.	Roast Temp, °C	Extraction, %	
		U ₃ O ₈	V ₂ O ₅
*573	None	98.2	69.3 (Optimum U ₃ O ₈)
*622	300	97.9	72.0
*621	400	97.9	72.0
*620	500	97.7	84.0
*628	600	97.4	82.7

After study, optimum conditions for acid leaching stockpile 28 ore were selected from the preceding series of tests. These conditions are:

- (1) acid - 700 pounds per ton
- (2) agitation time - 12 hours
- (3) grind - minus 16 mesh
- (4) density - 50% solids
- (5) temperature - 85°C

Five tests were run using the above conditions. Results are presented in Table XVII.

TABLE XVII

Optimum Leach Condition Tests

Test No.	Residue, %		% Soluble U ₃ O ₈ in Tails	Extraction, %	
	U ₃ O ₈	V ₂ O ₅		U ₃ O ₈	V ₂ O ₅
*578	.013	.13	--	96.7	82.7
*626	.007	.13	.001	98.2	82.7
*627	.008	.13	.001	97.9	82.7
40L	.013	.14	.004	96.4	81.3
41L	.010	.18	.001	97.4	76.5

Final test work on this ore was to determine the effect of sodium silicofluoride and manganese dioxide in hot acid leaching. Results of tests using silicofluoride are presented in Table XVIII.

This series of tests illustrates that vanadium recovery is somewhat improved by the use of silicofluoride and that uranium recovery is not adversely affected. The addition of 10 to 20 pounds of silicofluoride per ton of ore would recover about 10% more vanadium than under the optimum leaching conditions but the economics of such an expenditure are doubtful.

TABLE XVIII

Effect of Na_2SiF_6 on Stockpile 28 Ore

Conditions: Grind - minus 16 mesh
Density - 50% solids
Agitation Temperature - 85°C

Test No.	H_2SO_4 Lb/T	Na_2SiF_6 Lb/T	Agitation Time, Hr	Residue, %		Extraction, %	
				U_3O_8	V_2O_5	U_3O_8	V_2O_5
45L	500	10	12	.035	.26	91.0	65.4
46L	600	10	12	.012	.17	96.9	77.4
44L	700	10	6	.008	.05	98.0	93.4
48L	700	10	6	.007	.09	98.2	88.0
47L	700	10	12	.008	.12	98.0	84.0
49L	700	20	12	.008	.05	98.0	93.4

Tests performed by the Galigher Company using MnO_2 are presented in Table XIX.

TABLE XIX

Effect of MnO_2 (15 Lb/T) on Stockpile 28 Ore

Conditions: Grind - minus 16 mesh
Density - 50% solids
Agitation Temperature - 85°C
Agitation Time - 6 hr

Test No.	H_2SO_4 Lb/T	Extraction, %	
		U_3O_8	V_2O_5
636	500	97.2	65.3
637	600	97.4	74.7
638	700	94.6	78.7

Of all the oxidants used on Ute ore, only MnO_2 seemed to show an increase in uranium extraction. From the results above, it appears that 15 pounds of MnO_2 might replace 100 to 200 pounds of sulfuric acid per ton.

2. Exhaustion Studies - Utex Liquors

Thirty one different batches of Utex solution were run through the two-inch columns up to 7 June 1954. Analyses and cycles represented by each of the solutions are presented in Table XX. All of these solutions except Head number 9 were neutralized with hydrated lime before being run in the columns. The redox potential of all solutions made from stockpile 28 ore was suitable for ion-exchange and no reduction of solutions was required.

TABLE XX

Feed Liquor to IX Plant - Stockpile 28 Ore

Head No.	Resin Cycle Nos.	g/L			pH	emf mv	Solution to Ore Ratio
		U ₃ O ₈	V ₂ O ₅	SO ₄			
9	10C to 10D	1.88	4.38	57.0	1.26	-360	2.5:1 dilution
9A	11A to 14A	1.88	4.38	57.0	1.50	-355	"
10A	14B to 17D	2.30	4.37	53.5	1.70	-360	"
11A	18A to 19B	1.26	2.42	32.1	1.5	-360	"
12A	19C to 21D	1.33	2.52	34.8	1.5	-365	"
13A	22A to 24D	1.28	2.55	35.4	1.5	-365	"
14A	25A to 27C	1.30	2.44	37.3	1.5	-375	"
15A	27D to 29B	1.03	2.11	--	1.6	-400	"
16A	29C to 31B	1.15	2.04	--	1.5	-390	"
17A	31C to 33D	1.35	2.53	--	1.6	-380	"
18A	34A to 35D	1.27	2.32	24.7	1.6	-345	"
19A	36A to 38C	1.22	2.70	27.6	1.5	-380	"
20A	38D to 40C	1.35	3.39	--	1.5	-380	"
21A	40D to 42D	1.42	3.33	--	1.6	-370	"
22A	43A to 44C	1.57	3.28	--	1.7	-360	"
23A	44D to 47B	1.56	3.42	36.6	1.8	-365	"
24A	47C to 49D	1.40	2.84	38.3	1.6	-370	"
25A	50A to 52B	1.49	2.68	34.2	1.5	-350	"
26A	52C to 54B	1.38	2.34	29.2	1.5	-355	"
27A	54C to 57C	1.70	3.07	37.0	1.7	-350	"
28A	57D to 59D	1.22	2.04	25.1	1.6	-360	"
29A	60A to 61D	1.11	1.83	24.7	1.5	-370	"
30A	62A to 64B	1.14	2.14	22.2	1.7	-350	"
32A	64C to 66C	1.03	1.85	33.3	1.5	-370	"
33A	66D to 68D	1.14	1.94	26.0	1.8	-360	"
34A	69A to 68D	1.14	1.94	26.0	1.8	-360	"
35A	73A ₁ --	1.22	1.55	--	1.6	-390	"
35A	73A ₁ to 75A	--	--	--	--	--	"
36A ₂	75B to 75D	1.75	1.68	--	1.5	-380	"
36A ₃	76A to 76D	1.44	1.72	--	1.65	-370	"
37	77A to 77D	1.12	1.37	--	1.4	-375	"
38	78A to 79D	1.03	1.05	--	1.4	-390	"

Each of the two-inch columns contained 1500 ml of wet settled IRA-400 resin. This resin was new when placed in the columns but had been used for 10 resin cycles (40 loading cycles) on test work with tailings leach liquor prior to the time Utex liquor was fed to the units. While testing tailings liquor, an oxidized solution was run through the columns and vanadium loaded on the resin. At the end of 4 resin cycles on tailings liquor, the resin was cleaned with SO_2 and NaOH . The resin was not cleaned again before use on Utex liquors. Six resin cycles had therefore been completed on the columns when Utex studies were started.

Preliminary ion-exchange work was done in small, 25 ml single columns. This data indicated that Utex liquor would require a retention time of about 3 minutes and should have a pH value of 1.5. In actual practice in the two-inch units, retention times of from 3 to 6 minutes and liquor at a pH of from 1.4 to 1.8 were used. When the pH value of the feed liquor was below 1.4, breakthrough occurred much faster and as a result, saturation of the first column was not obtained. The same condition appeared when the pH value rose above 1.7. Within these limits, recoveries and loadings appeared to remain constant.

The relationship between retention time, cycle time and resin loading was studied on an early run using IX Head 9A. This head was produced by pugging and agitation and had a final dilution of 2.5 to 1. It was somewhat higher grade than the average solutions run in the unit as later work used liquors at a dilution of 3.0 to 1 to simulate practice in either RIP or CCD.

Retention times of 3, 5 and 6 minutes were used on this liquor. Differences in loading were slight, but noticeable. Cycle time (the time required to reach cutoff) was about 8.5 hours at 6 minutes retention time; about 7.5 hours at 5 minutes; and about 4.2 hours at 3 minutes. Because elution retention time was held near 12 minutes and the total time required for elution, backwash, displacement, etc., was about 7.0 hours, a 5 minute retention time was selected for this head. This allowed three cycles per day. The relationship between retention time, loadings, and resin cycle time is illustrated in Table XXI.

TABLE XXI

Retention Time vs. Resin Loadings

<u>Retention Time, Min</u>	<u>Loading</u>		<u>Cycles Per Day</u>	<u>lb U_3O_8 /cu. ft. /Day</u>
	<u>g/L</u>	<u>Lb/ft³</u>		
3	60	3.75	5.7	21.5
5	68	4.25	3.2	13.6
6	65	4.06	2.8	11.4

3. Exhaustion Data

A uranium balance for cycles completed through 20 April 1954 is presented in Table XXII. This balance includes all liquors fed to IX from Stockpile 28 pilot plant leaches and comprises 62 1/2 complete resin cycles (250 loading cycles).

TABLE XXII

IX Plant Balance - Utex Liquors

September 14, 1953 to April 20, 1954

<u>Product</u>	<u>Gallons</u>	Lb	Dist
		<u>U₃O₈</u>	<u>% U₃O₈</u>
Feed to Columns	4,661.64	52.996	100.00
Column Effluent	4,661.64	.100	.19
Absorbed by Columns		52.896	99.81
Eluates	845.11	51.316	96.83
Unaccountable (Recycle, Backwash, etc.)		1.680	3.17

Data for the cycles reported in the balance is presented in Table XXIII. These figures represent the overall average of all cycles and liquors run in the two-inch unit pertaining to Utex Ore.

TABLE XXIII

Data for Resin Cycles 10C to 73A

Utex Liquor Run - September 1953 to April 1954

Exhaustion

Head Solution:	pH	- 1.4 to 1.8
	emf	- 370 to -400
	g U_3O_8/L	- 1.36
	g V_2O_5/L	- c.a. 2.8
Flow:	Liters Throughout	- 17,646
	Time, Hours	- 1609.1
	Retention Time, Min-	3.3
Loading:	Average No. 1 Column to Saturation -	
	61.8 g U_3O_8/L WSR	
	3.86 lb $U_3O_8/cu. ft.$	
Barren:	Input, g U_3O_8	- 24,039.1
	U_3O_8 in Barren, g	- 45.46
	U_3O_8 in Barren, %	- 0.19
	Ave. Barren Comp. Assay, g U_3O_8/L	- 0.0026
Recovery from Solution:	99.81%	

Elution

Flow:	Liters to Precipitation	- 3,199.1
	Time, Hours	- 1,102.8
	Retention Time, Min, Ave.	- 12.4
Eluate:	Assay, g U_3O_8/L , Ave.	- 7.28
	g U_3O_8/L Eluted	- 23,277.0
	Assay, Final Eluate, Ave.	- c.a. 0.15 g U_3O_8/L

TABLE XXIV

IX Plant Balance - Utex Liquors

September 14, 1953 to June 7, 1954

<u>Product</u>	<u>Gallons</u>	<u>Lb.</u> <u>U₃O₈</u>	<u>%</u> <u>U₃O₈</u>
Feed to Columns	5,396.04	60.523	100.00
Column Effluent	5,396.04	.113	0.19
Absorbed by Columns		60.410	99.81
Eluates	947.18	57.914	95.87
Unaccountable (Recycle, Backwash, etc.)		2.496	4.12

Data for the cycles reported in the balance is presented in Table XXV. These figures are the overall average of all cycles and liquors run in the unit pertaining to Utex ore.

TABLE XXV

Data for Resin Cycles 10C to 79D

Utex Liquor Run - September 1953 to June 1954

Exhaustion

Head Solution:	pH	=	1.4 to 1.8
	emf	=	-370 to -400
	gms./liter U ₃ O ₈	=	1.34
	" V ₂ O ₅	=	ca. 2.8
Flow:	Liters Thruput	=	20,426.4
	Time, Hours	=	1,841.1
	Retention time, Minutes	=	3.24
Loading:	Average No. 1 Column to Saturation	=	
	63.0 gm. U ₃ O ₈ /L. w.s.r.		
	3.94 lb. U ₃ O ₈ /cu. ft.		
Barren:	Input, grams U ₃ O ₈	=	27,453.4
	U ₃ O ₈ in Barren, grams	=	51.24
	U ₃ O ₈ in Barren, percent	=	0.19
	Avg. Barren comp. Assay, g. U ₃ O ₈ /L.	=	0.0025
Recovery from Solution:	99.81 percent.		

Elution

Flow:	Liters to Precipitation	=	3,585.5
	Time, Hours	=	1,233.7
	Retention Time, Minutes, Avg.	=	12.4
Eluate:	Assay, gm./liter U ₃ O ₈ , Avg.	=	7.33
	Grams U ₃ O ₈ Eluted	=	26,269.8
	Assay, Final Eluate, Avg.	=	ca. 0.15 g. U ₃ O ₈ /L.

Two complete resin cleanups with caustic were made during the run on Utex ore and one minor cleanup was made when elution difficulties were encountered. The complete cleanups were made at the end of cycles 24 and 72. Although loadings had not shown any appreciable decrease at the end of cycle 24, the cleanup was made then to determine whether there were any contaminants on the resin. Some of the contaminants were probably due to tailings liquor that was run before the Utex liquor.

The cleanup at the end of cycle 24 was done by passing 5 liters of fresh, 10% sodium hydroxide through each column after first washing with water until a neutral pH was reached. This was followed by 2.5 liters of 1.0 molar chloride (as NaCl) and 1.5 liters of 10% hydrochloric acid. The caustic solution was assayed and results are shown in Table XXVI.

TABLE XXVI

Caustic Cleanup of Resin After 24 Cycles

Column	NaOH Eluate Assay, g/L					
	U ₃ O ₈	V ₂ O ₅	Mo.	P ₂ O ₅	Al ₂ O ₃	SiO ₂
A	.068	.26	.93	.20	.007	.32
B	.138	.16	.81	.24	.005	.32
C	.172	.15	.82	.24	.006	.30
D	.110	.16	.68	.25	.005	.20

The caustic analyses indicate that some uranium had not been eluted from the resin and was probably complexed with the phosphate which was also removed. Molybdenum was present on the resin to the extent of from 2.3 to 3.1 grams per liter of wet settled resin. While this is a visible loading, it had not affected uranium loading prior to the cleanup. Other ions removed were in minor amounts and had apparently little effect on uranium loadings.

Strong (1.0 Molar) hydrochloric acid was used to clean the resin after cycles 62 and 64.

At the end of Cycle 72, the resin was again cleaned with caustic. Resin losses were made up and the resin was treated with nitric acid to remove organic matter. This cleanup was accomplished similarly to the one after cycle 24 except that the nitrate wash was given the resin before conversion back to the chloride form. Results of this cleanup are presented in Table XXVII.

TABLE XXVII

Caustic Cleanup of Resin after 72 Cycles

Column	Vol 10% NaOH	NaOH Eluate Assay, g/L			Total g Mo	Mo Loading g/L WSR
		Mo	P ₂ O ₅	SiO ₂		
*A	9.5	3.13	.49	.4	29.75	19.8
*B	9.5	3.12	.53	.1	29.60	19.7
C	5.7	1.7	.53	1.2	9.69	6.4
D	5.3	4.1	.44	.2	21.73	15.1

*A and B columns retreated with caustic,
assay is average value.

Results of the second caustic cleanup show that molybdenum was building up on the resin. In the 48 cycles completed between the two cleanups, a little more than 0.5 gram of molybdenum was loaded per cycle. This is substantially all of the molybdenum present in Utex Liquor. Feed analysis shows molybdenum present in amounts from 0.02 to 0.06 grams per liter. Observation of the columns and results on exhaustion do not indicate that this molybdenum loading was totally responsible for the small drop in loading that was noticed prior to the second cleanup. Loadings had dropped from the average of 60 to 65 grams per liter of resin to 50 to 55 grams per liter just prior to the cleanup. The high sulfate eluting solution being used at the time was believed responsible for this drop but it may have been the molybdenum loaded on the resin that caused some of the decrease.

Present testing is being carried out utilizing liquors made by aqueous leaching at room temperature. Cycles completed to date show excellent loadings and no indication of molybdenum buildup. If periodic cleaning of the resin is required to remove molybdenum, it should not have to be done more than twice a year. This cleaning would not affect ion-exchange costs to any extent.

4. Elution Studies

Early elution practice at Monticello was limited to standard eluting solutions and techniques. As work progressed, various eluting schemes were tried in an effort to lower costs and increase efficiency. These general types of eluting solution were used. These were:

1. 0.9M NH₄Cl - 0.1M HCl, recycled with HCl makeup.
2. 1.0M NaCl acidified to pH 1.0 with H₂SO₄, no recycle.
3. 0.9M NH₄Cl - 0.1M HCl, recycled with HCl makeup and acidified to pH 1.0 with H₂SO₄.

The bulk of the elution studies was carried out with the third type of solution.

As the eluate was recycled according to scheme number 3, the sulfate ion concentration increased. A bleed was planned to eliminate sulfate ion buildup when and if elution efficiency dropped. However, instead of the expected drop, efficiency actually increased with continued recycle and no bleed was required. The sulfate ion concentration was allowed to increase to over 300 grams per liter.

Elution of the columns using 0.9M NH_4Cl - 0.1M HCl was fast and complete as shown by the elution curve for column 11B, Figure 3. Elution was complete in a little over 10 column volumes. Split elution could have been used to advantage at Monticello to lower elution costs. It was not done because loadings were of prime interest and it was believed that the data would be more reliable if series or split elution were used.

Elution using 1.0 molar NaCl acidified to pH 1.0 with sulfuric acid followed the curve shown for ammonium chloride elution. From a cost standpoint, it may be advisable to use NaCl eluting solution but the final product will be of a lower grade than when ammonium salts are used. Calcined product from ammonium chloride eluates averaged 94.5% U_3O_8 . Calcined product from sodium chloride eluates averaged 71.5% U_3O_8 .

After test work on the sodium chloride eluate was complete, a program of recycle of 0.9M NH_4Cl - 0.1M HCl eluting solutions was started. A short history of this program will aid in clarifying this technique.

On 26 October 1953, 40 liters of 0.9M NH_4Cl - 0.1M HCl eluting solution were made up. This solution was used to elute cycles 18C to 20D. It was used six times as a straight NH_4Cl - HCl solution, i.e., makeup HCl was added to bring chloride ion strength to 1.0 molar. After eluting cycles 21A and 21B, precipitation and makeup, the pH of the made-up eluant was 1.35. Since eluate volumes had been increasing from cycles 18C and 21B, sulfuric acid was added to the made-up eluant to bring the pH to 1.0, where it had been when the solution was freshly prepared. This batch of solution was used twice up through cycle 22B.

At this point, on 6 November, 1953, 40 liters of fresh NH_4Cl - HCl eluant was prepared. This was done to assure continuous operation by always having made-up eluant on hand for elution.

Cycles 22C and 22D were eluted with made-up solution (original 26 October solution). Cycles 23A and 23B were eluted with the fresh solution. From this point on until late in November, these solutions were admixed and used as one 80 liter lot of eluant. Sulfuric acid was added as necessary to maintain eluant pH at 1.0 after first making up chloride strength with HCl .

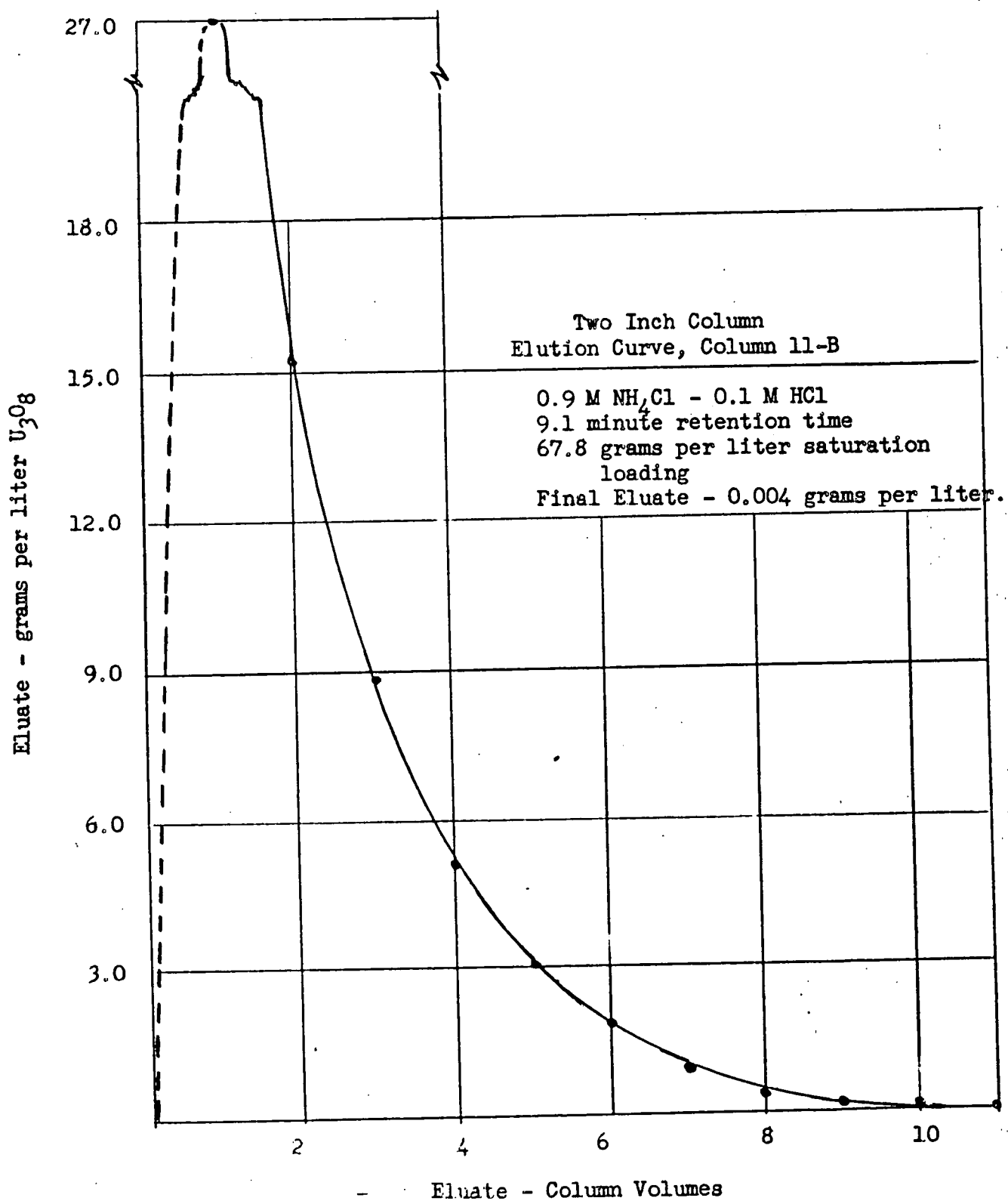


Figure 3

On 30 November 1953, it was evident that eluate volumes were decreasing substantially. Cycle 29B eluate was the first one checked for sulfate ion concentration. A sulfate concentration of 86.4 grams per liter was present at that time. As near as the records indicate, eluting solution at that time had been recycled a total of 13 times. At this time, the elution storage bottle fractured and about 20 liters of solution were lost. The remaining 60 liters of eluting solution were recycled up through resin cycle 64, which was run during March, 1954.

As of cycle 64, the eluting solution had been used (recycled) about 50 times and had been used for a total of about 200 elution cycles.

The sulfate ion concentration of cycle 64 eluant (solution made up to elute cycle 64 columns) was 302 grams per liter. The eluate volume required for cycle 64 averaged 7.0 column volumes per column.

Reagents added to this eluting solution during the period of its use averaged 2.0 pounds of 36% HCl and 3.0 pounds of 98% H_2SO_4 per pound of uranium eluted. Precipitation required an average of about 0.7 pound of anhydrous ammonia per pound of uranium precipitated.

The relationship between sulfate ion buildup in the eluting solution and eluting efficiency is shown in Table XXVIII.

TABLE XXVIII

Elution - Selected Cycles from 18 to 64

Column Eluted	Flow		Liters to Ppt	Eluate	Fin. El. g U ₃ O ₈ /L	Eluant g SO ₄ /L
	Time, Hours	Retention Time, Min		g U ₃ O ₈ /L		
18A	5.7	11.0	18.5	4.50	--	--
B	2.8	5.4	18.4	4.58	.2	--
20C	4.0	6.9	21.0	4.30	.2	--
D	4.0	7.2	20.0	4.24	.2	--
21A	5.1	9.4	19.5	4.63	.15	--
C	5.0	11.5	18.5	4.5	.27	--
26A	4.1	10.5	14.0	6.03	--	--
C	4.7	9.9	17.0	4.85	.05	--
29A	3.9	9.7	14.5	6.92	.20	* 86.4
D	3.7	9.9	13.3	7.33	.24	"
30B	4.3	13.0	13.0	7.63	.10	*111.1
31B	3.8	10.8	11.5	8.90	.13	--
34B	4.1	12.4	11.8	9.43	.33	165.9
35B	3.8	13.0	10.6	10.82	.40	*183.1
39C	4.1	12.3	10.2	8.75	.17	206.6
42C	4.0	13.5	10.7	9.46	.10	214.8
45D	4.0	13.2	10.9	9.43	.25	224.7
47C	4.08	15.0	9.8	9.01	--	234.2
50C	3.25	12.2	9.6	9.16	.14	245.7
58B	4.00	13.7	10.5	8.30	.25	286.4
64D	4.50	15.0	10.8	6.16	.35	302.1

* SO_4 in eluate.

These data show that the eluate volume decreased as the sulfate ion concentration of the eluant increased. A plateau was reached when sulfate ion concentration reached 150 to 175 grams per liter and continued until the concentration reached over 300 grams per liter. There is a possibility that the plateau extends to concentrations greater than 300 grams of sulfate per liter.

It may be seen from Table XXVIII that the volume of eluant required for complete elution decreased from 18.5 liters when the solution was new to 10.0 to 11.0 liters when the sulfate concentration was about 165 grams per liter. Eluate volumes for cycle 20 were increased to 21.0 liters, probably due to a slight buildup in sulfate ion concentration.

As far as elution is concerned, there is no indication that a buildup of sulfate ion concentration beyond 300 grams per liter is harmful. Elution of columns at cycle 64 was as good as at cycle 34 or 35. However, resin loadings had dropped about 15% from cycles 53 to 62. To determine the cause of this drop, the resin was eluted with 1.0 molar HCl at the end of cycle 62. Before elution with HCl, the resin was removed from the columns, washed by decantation, screened through a 16 mesh screen, and measured before returning to the columns. Data for this resin cleanup are presented in Table XXIX.

TABLE XXIX

HCl Resin Cleanup after 62 Cycles

<u>Column</u>	<u>Resin Recovered ml. WSR</u>	<u>HCl Eluate</u>			<u>Sulfate Loading g/L WSR</u>
		<u>Liters</u>	<u>g/L Sulfate</u>	<u>Gms. Sulfate</u>	
A	*1350	5.2	19.0	98.5	73.0
B	1380	5.4	10.0	54.0	39.0
C	1390	5.2	9.9	51.6	37.1
D	1380	5.0	9.8	49.2	35.7

* 1500 ml. originally added to each column

There was an insignificant amount of uranium present in the HCl eluate. After the cleanup, resin capacity went back to normal loadings of about 60 to 65 grams per liter of wet settled resin for one cycle (cycle 63). Loadings for cycle 64 again dropped off and were erratic. Eluant used to elute cycles 63 and 64 contained about 310 grams sulfate per liter.

At the completion of cycle 64, resin was again eluted with fresh 1.0 molar HCl with results similar to those obtained after cycle 62. Data for this cleanup are presented in Table XXX.

TABLE XXX

HCl Resin Cleanup after Cycle 64

<u>Column</u>	<u>HCl Eluate</u>		<u>gms Sulfate</u>	<u>Sulfate Loading g WSR/L</u>
	<u>Liters</u>	<u>g/L Sulfate</u>		
A	5.0	6.2	31.0	23.0
B	10.3	37.4	385.0	279.0
C	11.1	5.8	64.4	46.3
D	9.8	7.7	75.5	54.7

The high sulfate loading for column "B" has been checked and appears to be correct. There is no explanation here as to why the column should take up more sulfate than the others.

A sample of cycle 54 eluant was given to Mr. Dan McLean who took it to the Bureau of Mines in Salt Lake City for analysis. Results of that analysis are shown in Table XXXI.

TABLE XXXI

Cycle 54 Eluant Analysis

<u>Cl</u>	<u>SO₄</u>	<u>NH₃</u>	<u>CaO</u>	<u>MgO</u>	<u>H₂SO₄</u>
35.7	279.6	65.0	.2	.3	31.8

Since it was possible that the recycle eluant was responsible for lowered resin capacity, the recycled solution was reserved and fresh eluant made up. New eluant was made up to the concentration of recycled eluant that gave the best results. Solution was made up to 0.9 molar in NH_4Cl , 0.1 molar in HCl , with sulfate ion strength at 150 grams per liter as ammonium sulfate. This eluting solution has been used since cycle 64 with good results. About a 10% bleed is maintained on the eluting solution to keep the sulfate concentration at near 150 grams per liter. Loadings are up to 65 grams U_3O_8 per liter of resin and eluate volumes are running about 7.0 column volumes for complete elution.

Much work remains to be done on determining which factors affect elution efficiency. Present testing of Utex liquors using an eluting solution containing 150 grams of sulfate per liter is encouraging. Use of recycle solution on clean resin may show whether the high sulfate concentration was responsible for decreased loadings or whether it was the molybdenum loading, or a pH change in the leach liquor resulting from a faulty pH meter, that caused the drop in loadings.

C. Stockpiles 19, 24, 29, 6, 16 -

Test work on stockpiles 6 and 16 is incomplete at this writing and will be reported at a later date.

Table XXXII presents results obtained when liquors produced by bench scale testing were ion-exchanged. Some of the saturation loading data is incomplete because of insufficient solution to saturate the columns. All liquors were adjusted to a pH of 1.4 to 1.7 and reduced to at least -425 mv. before being run.

These data show that Temple Mountain roasted ore liquors were more amenable to ion-exchange than raw ore liquors. Five minute retention time gave higher loadings (51.3 vs. 39.2 breakthrough loading) than 3 minutes on this liquor.

Stockpile 16, mostly Luckachukai type ores, showed satisfactory loading of 48.2 gms. U_3O_8 /liter w.s.r. This liquor required reduction with iron.

Table XXXII

25 Ml. Column Results - Various Monticello Stockpiles
All Tests at 3 Minute Retention Time unless Noted.

Test No.	Ore	Analysis			pH	emf	LOADING		Saturation	C.V.	C.V.	Elution	
		U ₃ O ₈	V ₂ O ₅	Mo			Breakthru	g/l				Assay U ₃ O ₈	Fin El. U ₃ O ₈
												g/l	g/l
15X	Temple Mount. - Raw	1.09	2.37	--	1.55	-325 mv.	44	48.0	88	52.9	13.6	3.89	.21
17Y	" " Roast	1.40	3.07	--	1.4	-390	28	39.2	68	75.4	12.4	6.07	.19
16X	Stockpile 16 ^{/1}	1.20	3.49	.007	1.5	-320	26	31.2	71	48.2	12.7	3.78	.01
^{/2} 17X	Muddy River	.93	.27	.10	1.5	-425	10	9.3	40	9.0	10.3	.87	.20
^{/3} 18Y	Slick Rock	1.70	6.93	.028	1.7	-410	16	27.2	42	17.5	11.2	1.56	.05
^{/4} 19Y	Temple Mount, Roast ^{/1}	1.22	3.37	--	1.5	-380	42	51.3	--	63.2 ^{/5}	10.9	5.81	.13
18X	Stockpile 19 ^{/1}	.75	.26	--	1.45	-380	30	22.5	68	35.0	9.7	3.60	.14
20Y	Stockpile 26 ^{/1}	.75	.18	--	1.45	-390	52	39.0	--	46.5 ^{/6}	12.0	3.86	.13
19X	Stockpile 6	.99	6.16	--	1.5	-375	42	41.6	78	52.6	12.4	4.23	.16

Notes:

- ^{/1} Liquors reduced with iron nails before neutralization.
- ^{/2} Incomplete Elution - Column cleaned with NaOH. NaOH El. (6.7 C.V.) ran 0.151 g/l U₃O₈; 0.192 g/l Mo.
- ^{/3} Incomplete Elution - No data taken on cleanup.
- ^{/4} 5 Minute Retention Time
- ^{/5} Not Saturated - Loading is for 40 % saturation.
- ^{/6} Not Saturated - Loading is for 56 % saturation.

Stockpiles 19, 26 and 6 all produced liquors that appeared amenable to ion-exchange. Stockpile 29 (in this series of tests) and a sample of Muddy River ore did not give satisfactory ion-exchange results.

The data presented in Table XXXII, indicate that all of the ores presently stockpiled at Monticello are amenable to ion-exchange and acid leaching. Muddy River ore is not presently stockpiled at Monticello, so no further work was done on it.

Column Data

Data for the 10 resin cycles completed on Stockpiles 19, 24 and 29 in the 1/2-inch columns is presented in Table XXXIII.

TABLE XXXIII

1/2-Inch Unit - Cycles 1 to 10
Stockpiles 19, 24, 29 Liquors

EXHAUSTION

	19	24	29
Head Solution: pH	= 1.4 to 2.0	1.6	1.4 to 1.6
emf	= -380	-400	-400
g./L. U_3O_8	= 1.05	1.37	1.20
g./L. V_2O_5	= .36	ca. 2.50	2.83
Flow: Liters Thruput	= 41.065	39.182	43.345
Time, Hours	= 295.82	231.68	282.89
Ret. Time, Mins. Avg.	= 4.32	3.55	3.36
Loading: Avg. #1 Column to Saturation, mg./ml.	= 50.4	64.2	65.6
Barren: Input, U_3O_8 , grams	= 43.190	53.58	51.965
Loss, Displacement	= 2.744	3.70	3.051
Net Input, grams	= 40.446	49.88	48.914
U_3O_8 in Barren, grams	= .207	.137	.176
U_3O_8 in Barren, percent	= .51	.26	.36
Avg. Barren Comp. Assay, g./L.	= .005	.003	.004
Recovery from Solution:	99.49%	99.74%	99.64%

ELUTION

Flow: Liters to Precipitation	= 13.950	13.838	13.209
Time, Hours	= 154.8	180.00	158.82
Ret. Time, Mins. Avg.	= 6.66	7.80	7.21
Eluate: Assay, gm. U_3O_8 /L., Avg.	= 2.65	3.13	3.60
Grams U_3O_8 Eluted	= 36.932	43.34	47.525
Assay Final Eluate, Avg.	= ca. .20	ca. .15	ca. .15

Aside from rather high eluate volumes, probably due to short elution retention times, these three stockpiles show good amenability to ion-exchange. No difficulty should be encountered if they are treated by ion-exchange.

ACKNOWLEDGEMENT

All of the work reported here was done with the cooperation of the Galigher Company and grateful acknowledgement is here given of their help and consideration.

Mr. Helmer A. Johnson, Resident Manager; Mr. J. W. Joyce, Plant Superintendent; Mr. D. E. Poormen, Plant Metallurgist and his staff; and Mr. B. D. Miller, Chief Chemist and his staff have all contributed willingly and wholeheartedly to the test work program on Utex ores.

Some of the work, especially that pertaining to Utex ores, was carried on cooperatively with the Galigher Company and testwork attributed to them is reproduced with the permission of Mr. Johnson.

The writer also wishes to commend Mr. Alex Jameson, Galigher Company Laboratory Assistant who was assigned to this project at its inception. His attention to detail and his help in the program have been invaluable. He has shown a natural aptitude for the work and his interest in the project has aided us many times.